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different RL's are used in the blend. The calculations took into account the efficiencies of the various tobacco producing processes and held tobacco input into Philip Morris constant. The calculations showed that if reaction flavored RL is substituted for current production RL in Marlboro that the amount of tobacco feed required increases if cigarette OV is maintained. If cigarette RH is held constant, however, the tobacco feed required is the same for both RL's. Holding blend RH constant results in lowering pack OV ≈0.30%.

mkb

Christopher L. Lig

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B. Stockton Street Plant--Infestation Inspection

Cigarette filler in saratogas was found to harbor adult beetles. Appropriate control steps were recommended.[5]

IV. REFERENCES

1. Lehman, R. M. Notebook No. 7235, p. 99.
2. Lehman, R. M. Notebook No. 7235, p. 100.
3. Long, J. S. Notebook No. 7283, p. 113.
4. Long, J. S. Notebook No. 7283, p. 114.
5. Lehman, R. M. Notebook No. 7235, pp. 93-95.

Art Magaletti

np

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REFERENCES

1. "Tobacco Leaf Curling," J. C. Crump, III and Linda Trentham to Karl Jockel and Frank Lipski.

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V. Services to Other Groups

Flavor revisions were made in the "Lolita" aftercut formulation for PM Europe.

Subjective screenings of cigarettes from Project "Mild" are in progress.

Subjective evaluations were conducted on various adhesives in Merit, Marlboro and Saratoga cigarettes and plasticizer samples were evaluated on Marlboro cigarettes.

VI. References

Notebook	Pages
7282	196-199
7352	190-196
7427	62- 65
7453	186-200
7522	1- 10
7527	27- 48
7560	1- 41

William R. Bell

/ds

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Source: https://www.industrydocuments.ucsf.edu/docs/sjjm0000

REFERENCES

1) B. E. Waymack, Notebooks 7482 and 7562.
2) C. O. Tiller, Notebook 7529.
3) P. E. Phillips, Notebook 7500.
4) P. A. Wilson, Notebook 7365.
5) D. B. Losee, Notebook 7006.
6) K. R. Squire, Notebook 7440.
7) K. A. Cox, Notebook 7507.
8) D. L. Simpson, Notebook 7141.
9) J. F. Bebbs, Notebook 7449.
10) H. A. Hartung and K. R. Squire, "IBM 5120 Computer System," memo to John Higgs, July 18, 1980.

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weight and the percent composition of the polymer in each sample. Memos reporting the results have been sent to the requestor (H. Grubbs).

C B Hoelzel

/ev

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CHARGE NUMBER: Various

PROGRAM TITLE: Analytical Research

WRITTEN BY: W. R. Harvey, D. C. Watson, and R. E. Davis

DATE: August 11, 1980

I. TIPPING PAPER INK/ADHESIVE PROBLEM

Work on the Ferrox stain and critical surface tension experiments was presented to the tipping paper/adhesives task force. Correlation of these experiments to machine performance and performance of the several variables represented in the tipping paper prepared at Milprint are still pending.

Identification work is continuing on several as yet unknown peaks found in the HPLC of ink extracts.

II. QUALITATIVE TEST OF NITROCELLULOSE ON WHITE TIPPING PAPER

The reagents were prepared and a qualitative test for nitrocellulose was demonstrated for the Brand Modification Group. The reagent was given to them for their use in the qualitative identification of nitrocellulose on white tipping paper.

III. AGING EFFECT OF KJELDAHL TOTAL NITROGEN STANDARDS

The study of the aging of the total nitrogen standards in glass and Nalgene® storage has been completed. By statistical evaluation it was demonstrated that Nalgene® storage is stable over the 51-day period of the study whereas glass storage is not.

IV. NO_2 SAMPLE MANAGEMENT (PROJECT NO. 1752)

A series of programs have been designed, written and debugged to aid in the analysis, interpretation and report generation for the NO_2 environmental study. The programs fall into three distinct categories.

1. Acquisition of data from the AutoAnalyzer by MINC-11 computer. Sample ID is input by a interleaved 2 of 5 bar code. Additional program features include least square standard curve fit, rejection of outliers, signal averaging and storage of final and intermediate results on diskette.
2. VIP input files are built by a set of programs that are designed to read data from questionaires and logs by using a graphics tablet and from data received from the MINC-11 computer.
3. VIP (Versatile Information Processor), a data base management system, will be used to correlate data and produce desired reports.

W. R. Harvey

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III. RCB Studies

It has previously been reported that the RCB samples produced in the laboratory are four to five times stronger than the BL production material and that the laboratory samples containing standard amount of stems are twice as strong as the similarly processed samples that did not contain stems.[5]

This study was then followed with production RCB slurries which were cast in the laboratory at the standard 17.5% solids and over a range of 8-15% solids. The sheets that made at the lower solids were found to be the strongest (2.2 kg/in with 8% solids vs 1.3 kg/in with 15% solids).[5] This indicates that the BL production slurry is sufficiently refined and the problem is probably due to the inability of the high solids slurries to "heal" the deformities caused during drying. This phenomenon is expected to be magnified by the production method of drying (forced hot air circulation) which causes a pH change in the cast slurry by removing NH_3. These studies are being continued in order to produce the proper samples with approximately production conditions prior to recommending changes for evaluation at the BL PP, and in production. In the meantime, an effort is also being made to determine the minimum levels of stems or other additives that are needed to produce acceptable RCB.

IV. References

1. Susan Evans Notebook No. 7318 pp 96, 100, 101, 103

2. H. A. Jones Notebook No. 7347 p 165

3. Susan Evans Notebook No. 7318 pp 102, 104-107, 109, 112-113

4. H. A. Jones Notebook No. 7347 pp 161-163, 166

5. "Monthly Progress Reports", June 1-30, 1980 under charge No. 1503

6. J. W. Leik Notebook No. 7395 pp 90-93, 102, 103 and 105

G. D. Kuitse

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TABLE I - % Reduction in Sample Area After Treatments

	66% RH 13.7% OV	73% RH 16.7% OV	76% RH 17.6% OV	80% RH 21.9% OV	92% RH 40.7% OV
Before Heating	9.2%	11.3%	11.5%	11.3%	11.9%
125°F	13.6%	15.0%	29.2%	23.0%	27.3%
150°F	14.3%	20.3%	25.2%	20.0%	23.3%
175°F	12.6%	17.5%	26.0%	27.0%	34.1%
200°F	12.4%	19.2%	24.2%	23.0%	32.0%
225°F	11.8%	15.3%	21.4%	23.0%	31.7%

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-The only crystalline component found in samples of material from the crystallizer of the denitrification process at Park 500 was potassium nitrate.

Mary Ellen Counts

/jsa

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CHARGE NO: 1702

PROJECT TITLE: FILTRATION PHYSICS

PROJECT LEADER: R. W. Dwyer

PERIOD COVERED: July 1-31, 1980

DATE OF REPORT: August 6, 1980

I. FILTER DESIGN

A. FILTER EFFICIENCIES (1,2)

The filtration-efficiency studies have been extended to an aerosol flow rate of 5000 cc/min for 2.1/60K cellulose acetate filters. These data show that the efficiency increases in the range of 1300 to 5000 cc/min, suggesting that the inertial removal mechanism makes a significant contribution at these flow rates. Preliminary experiments with a 2.5/48K tow show a similar dependence of efficiency on aerosol velocity.

B. DESIGN MODELLING (3,4)

A report was issued on the effects of design on flow through ventilated cigarettes. Further work has been performed on refining the filtration-efficiency section of the model in an attempt to predict puff-by-puff TPM deliveries at elevated flow rates.

C. NOVEL FILTER DESIGNS (5,2)

The Flavor Development Panel subjectively evaluated our hollow-tipped filter cigarettes compared to untipped controls. The prototype filter was designed to provide more TPM than the control at the early puffs, and less at the later puffs. The Panel found no subjective advantage at the 5 mg level. The C.I. puff-by-puff analysis of these cigarettes has not yet been received.

FTC tar results have been obtained from C.I. on our filter by-pass samples. These cigarettes are being redesigned to give deliveries comparable to unmodified controls.

A program has been initiated to determine the effects of dilution on the deliveries and filtration efficiencies of non-standard, commercially available cigarette filters. Baseline results for Merit 85's have been received, and

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CHARGE NUMBER: 1101

PROGRAM TITLE: ENTOMOLOGICAL RESEARCH

PERIOD COVERED: July 1-31, 1980

PROJECT LEADER: M. A. Manzelli

DATE OF REPORT: August 6, 1980

I. INSECT GROWTH REGULATOR

A. 1976 Bright Strip and Stem KABAT® Application

The few remaining hogsheads of this treatment (retained in the miniwarehouse study) have been sampled for final bio and chemical assays.[1]

B. Commercial Application of KABAT®

A seminar on KABAT® usage was presented to the technical staff of the Universal Leaf Company, members of which will be involved in the application of the IGR to PM tobacco.

II. PHYSIOLOGY OF THE CIGARETTE BEETLE

A. Effect of Phosphoric Acid on Growth Cycle

Eggs placed in tobacco treated with 0.1, 0.25, 0.5, and 1.0% H_3PO_4 have hatched and given rise to adults.[2]

B. Nonacclimated Exposure to -6.7°C (20°F)

The third and final replicate has been completed in this study. A summary of the three series of data shows the following mortality times (in days) for the four beetle stages at -6.7°C exposure:

Adults	Pupae	Larvae	Eggs
3	4	4	7

The next series will involve the same temperature exposure, but will be preceded by exposing the four beetle forms to 12.5°C (55°F) for seven days (acclimitization).[3]

III. ASSISTANCE TO AREAS OUTSIDE R & D

A. Costa Rica KABAT® Study

Samples of KABAT® treated tobacco from Costa Rica are being bioassayed to check analytical findings.[4]

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July 1 - 31, 1980

Date Issued: August 15, 1980

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III. Denitration

The pilot plant electrodialysis unit was run continuously from 7/7/80 to 7/24/80 to accumulate sufficient running hours for Ionics Inc. to establish an estimate of membrane life. The total cumulative running time at the end of this run was 20 days. The volumne of liquor (18% HWS-split flow mode) denitrated was 17,000 gallons. The recirculation rate (100 gal/min.) through the membrane stack was reduced by 15% each day but was restored almost to its original value by water flushing. On opening the stack for inspection an accumulation of calcuim phosphate was found at the cathode in both the process and the brine streams and throughout the pathways of the membrane spacers. A thorough discussion was held with Ionics about the performance of the ED unit to date. They thought the membranes were not ionically fouled and would have an average life of at least six months. The mechanical fouling of the pathways in the stack was thought to be caused by the cathode not being electronically insulated from the liquor streams. New insulated cathodes were installed in the stack and another continuous denitration run was begun on 6/8/80 after acid washing the membranes in situ to regenerate their activity. After five days of operation the recirculation rate through the stack was reduced by less 10% (<2%/day) and a satisfactory denitration rate was being maintained. Technical analysis by the ED pilot plant operation by ourselves and Ionics will be completed by mid-September and a cost estimate for Park-500 completed by mid-October.

[1]Park-500 steering Committee History #3 7/30/80.

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tower.

Tower exit tobacco samples expanded using four different processes and examined as a function of time using both EPR and diffuse reflectance spectroscopy have revealed an increase in the moisture content of the samples over a 30 day period and a plateau in the number of matrix stabilized radicals after approximately 16 days. Equilibrated samples displayed an insignificant variation in the radical content in this same period.

MATHEMATICAL MODEL OF A BURNING CIGARETTE (6,7,8)

The recent installation of an HP plotter in Building 36 by the Computer Application Division and their subsequent support has greatly improved our on-site computing capabilities. Much effort has gone into graphing in standard formats all of the relevant data thus far acquired on the pyrolysis and combustion of bright tobacco with the aim toward assembling a coherent "picture" of the thermal decomposition processes involved.

A computer program has been written which simulates the flow of an aerosol around an array of cylinders using a modified flow field which accounts for the slip of the gas as it flows by the cylinder. Comparison of these results with an alternative approach used in the literature will provide a check on the simulation procedure.

In cooperation with Project 1702 a procedure was developed for determining the size distribution at a number of wavelengths.

The new filtration model for calculating the filtration efficiency of a polydisperse aerosol from the filtration properties of the individual particle sizes was reformulated in more generalized form allowing more freedom in its use.

SPECIAL TOPICS (9,10)

Documentation of output options for workspace and file management in the APL Account 79 modelling system is nearly complete.

Work on the current phase of the Westab modelling project has been completed.

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CHARGE NUMBER: 1005

PROJECT TITLE: Improved Semiworks Operations

PROJECT LEADER: J. F. Sherwood

PERIOD COVERED: July, 1980

DATE OF REPORT: August 7, 1980

I. Primary Processing

A. WS Application

A series of tests was completed in the MC to confirm the previous bench scale tests which indicated that it is feasible to apply WS to tobacco in the MC, using the proposed covered and exhausted conveyor system, if a reduced tobacco rate (4,000 lb/hr) and a concentrated WS solution (24 weight % in ethanol) were used. In these experiments, B&H Menthol after-cut containing the normal amount of ethanol as well as 1.5 and 2.5 times the normal amount was applied to B&H filler in the MC at tobacco rates of 4,000 lb/hr. This would result in theoretical ethanol levels on the filler ranging from 1.2 to 3.0%. The theoretical ethanol level for filler to which WS and a special Merit after-cut are applied is 2.5%. It was assumed in these tests that the existing covered and exhausted conveyors on the MC menthol line would approximate the ethanol removal of the proposed conveyor system for WS application.

The results of measurements made with an MSA combustible gas indicator on tobacco samples collected after the final weigh belt showed ethanol levels in the air over the tobacco to average 2, 2, and 4% of the LEL (lower explosive limit), respectively, for the samples with the normal amount, 1.5 times the normal amount, and 2.5 times the normal amount of ethanol. Another run made at an 8,000 lb/hr tobacco rate using 2.5 times the normal amount of ethanol resulted in an average ethanol level in the air over the tobacco samples of 10% of the LEL. Therefore, it appears very likely that WS test market runs at 8,000 lb/hr will be feasible in the MC.

B. Filler Degradation

As part of a program to determine if individual components, particularly RL, degrade more than others in a cigarette blend, an experiment was run in which an improved urea analysis procedure, developed by R. E. Davis, B. M. Handy, and A. B. Smiley, was used to trace RL in the individual sieve fractions of Marlboro filler. In this experiment, blend sieve fractions before and after a Mark 9 maker were analyzed for urea. Analysis of variance of the data showed significant differences in the urea levels in the individual sieve fractions with the mediums fractions showing the highest urea levels. However, there were no significant differences in the urea levels before and after the maker indicating that the RL did not degrade any more than the remaining portion of the blend.

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samples of True, Kent III, and L & M Flavor Lights are being prepared.

II. AEROSOL CONTROL

A. FILTRATION EFFECTS (6,7,8)

A report was issued on the effects of puff velocity, rod lengths, and filters on smoke formation and particle size. A thorough investigation of the influence of rod length and aerosol flow rate on the efficiency of Marlboro tobacco rods is under way. In this study, the efficiency of the tobacco bed in selectively removing particles of a specific size is being examined.

A dual-wavelength light-extinction apparatus has been constructed and made operable. This instrument will be used to determine the feasibility of monitoring the particle size changes in smoke exiting the filter during a two-second puff.

B. PARTICLE SIZE ANALYSIS (9)

A Sierra Instruments Cascade Impactor arrived this month. This device should allow us to achieve higher resolution in the particle size analysis of smoke aerosols. Currently, the impactor is being calibrated.

C. REFRACTIVE INDEX ANALYSIS (10)

A report was issued on the refractive index of cigarette smoke.

REFERENCES

R.W. Dwyer

1) S. G. Abel, Notebook 7516.
2) R. Tyson, Notebook 7543.
3) R. W. Dwyer, Notebook 7327.
4) R. W. Dwyer, S. G. Abel, M. L. Fleming, special report, "The Effects of Design on Flow through Ventilated Cigarettes," A.N. 80-172, 7/2/80.
5) M. L. Fleming, Notebook 7342.
6) R. M. Creamer, Notebook 7237.
7) D. M. Puzio, Notebook 7553.
8) R. M. Creamer, special report, "The Effects of Puff Velocity, Rod Length, and Filters on Smoke Formation and Particle Size," A. N. 80-183, 7/20/80.
9) D. D. McRae, Notebook 7330.
10) D. D. McRae, special report, "The Refractive Index of Cigarette Smoke," A.N. 80-190, 7/17/80.

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CHARGE NUMBER: 1600

PROJECT TITLE: Smoker Psychology

PERIOD COVERED: July 1-31, 1980

PROJECT LEADER: W. L. Dunn

DATE OF REPORT: August 12, 1980

Project Title: Nicotine in Saliva
Written by: F. J. Ryan

A gas chromatograph is being installed in the lab to enable us to make salivary nicotine measures. The procedures to be used are described in Special Report 80-198 by Don Magin and George Walker.

Project Title: Firmness Evaluation Training
Written by: F. J. Ryan

A panel of 11 employees has been selected to begin judging the firmness of unlit and lit cigarettes. The panelists are generally able to detect firmness differences between test cigarettes weighing .91, .95, .98, 1.03, and 1.07 g. A cigarette firmness rating scale ranging from 20 (least firm) to 40 (most firm) has been developed, and panelists are learning to associate numbers to the firmness sensations from the test cigarettes.

Project Title: Nicotine Discrimination at 16 mg Tar
Written by: F. J. Ryan

Data gathering continues. We have complete data from 12 subjects and incomplete data from 8 more.

An Annual Report was written this month.

W L Dunn

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II. Cigarette Making and Packing

High Speed Seam Sealing

POL results for Marlboro smokers on Marlboro (N-3121) cigarettes made with Polymer Industries 428-156-1 PVA liquid adhesive showed no significant differences from control cigarettes made with starch paste. However, significant differences were detected between the test and control by non-Marlboro full flavor smokers. Plans are to test another lot of the Polymer adhesive.

III. Blend Component Studies

Expanded Thin Bright and Burley

A test is in progress to evaluate the expansion of thin bright and thin burley tobacco. In this test, run in the MC, two expansion runs each were made using bodied and thin bright and burley tobacco. CV results showed the increase in filling power due to expansion to be 38.3 and 43.2 cc/10g for the bodied and thin bright, respectively. The CV increases for the bodied and thin burley were 43.9 and 46.0 cc/10g, respectively.

Cigarettes have been made from these samples and firmness as well as analytical testing is in progress. Subjective testing by the Flavor Development group should begin next week on Marlboro type cigarettes having the following types of expanded tobacco at a 10% level: DBC bright, thin bright, thin burley, and 50% thin bright/50% thin burley.

J. F. Sherwood

J. F. Sherwood

mkb

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CHARGE NUMBER: 0307

PROJECT TITLE: Cigarette and Tobacco Measurement Methods

PROJECT LEADER: C. L. Irving

PERIOD COVERED: July, 1980

DATE OF REPORT: August 7, 1980

I. Moisture Measurement

The automatic microwave moisture meter was received from Steinbrecher Corporation, checked out and set up for testing. Initial testing of the L-band unit, which uses 5 to 25 gram tobacco samples, will be done with Marlboro and Merit cut filler at seven different OV levels. The data from these tests will be analysed by Mr. J. E. Tindall to determine the amount of variation in the OV test relative to the microwave test. The data from this test will also be used to generate an empirical relationship to predict OV. The X-band unit, which uses cigarette rods, will be tested statically in the laboratory before mounting the unit on a Mark 9 for dynamic tests. The data analysis procedure for the X-band unit will be like that used on the L-band unit. Preliminary tests conducted with Dr. Steinbrecher showed a correlation coefficient of 0.997 between microwave attenuation and OV for 15 gram Marlboro samples. The more exhaustive testing outlined above is now underway.

II. Cigarette Firmness

The cigarettes made for the subjective evaluation of the firmness while smoking characteristics of cigarettes were machine tested. Frank Ryan has begun subjective testing and is currently teaching panelists to assign numerical values to the cigarettes of different firmnesses. Thus far no problems have been encountered by the panelists when ranking cigarettes from softest to firmest. Once the panelists have been trained, cigarettes will be partially smoked by machine and the panelists will be asked to rank them by firmness. This phase should begin in about two weeks.

III. Dynamic Circumference Transducer

Additional testing of the strain gauge spring dynamic circumference transducer on a Mark 8 maker showed approximately 50% of the variation observed in dynamic tests to be due to short term drift of the system. This short term drift is most likely caused by small mechanical changes in the system or amplifier drift. The mechanical rigidity of the system should be improved when the unit is moved to the Mark 9 due to the improved bracket design. If short term drift remains a problem after that, semiconductor strain gauges will be used to replace the constantan gauges currently used. The semiconductor strain gauges have a much higher output for a given strain, allowing the amplifier gain and its associated noise to be reduced.

IV. Mathematical Modeling

The mathematical modeling routines were used to evaluate the effect of RL filling power changes on tobacco utilization. The calculations used current production Marlboro as a baseline to predict the tobacco required per 1000 cigarettes if

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Tests are planned on clumps which will be exposed to a "free fall" on pins in a rotary cylinder. Similar tests are scheduled with flights as the breaking mechanism. Attempts are being made to locate shakers with a big thrust which could cleave the clumps along their fracture plane without any cutting action.

C. Accelerated Drying of RL by Microwaves (P. Aument, R. Burde)

A second series of tests on filler and RL samples subjected to a rapid and extensive overdrying is in progress. The 15 KW unit at Finessa is being used.

V. Patent Activities

The final draft of the application on the electrostatic separation of tobacco from stemmery discards was reviewed, corrected and signed by all proposed inventors. The application on the ammonium carbamate reaction nozzle was also completed. The DIET interference trial has been scheduled for September 29.

[1]Notebook 7414, pp 35-48.

J V Utsch/deb

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CHARGE NUMBERS: Various

PROJECT TITLE: Flavor Development

PROJECT LEADER: C. N. Kounnas

PERIOD COVERED: July, 1980

DATE OF REPORT: August 5, 1980

WRITTEN BY: William R. Bell

I. Low Delivery Program

Testing of the 100mm Cambridge Menthol Cigarettes has been requested for four (4) models vs. Carlton 100mm menthol cigarettes. These cigarettes will be made in early August, 1980.

The Merit Ultra Lights (MUL) program is continuing with POL and VP testing. Menthol candidates have been made and are being evaluated.

II. Aftercut Flavor and Casing Modification Program

POLs 4136, 4137, and VP 4750 are awaiting cigarettes. POL 3105 is pending remake. POL 4114 is pending shipment. POLs 3090, 3098, and 3099 are in the field. Preliminary results on POLs 3091, 3102, and 3103 have indicated no significant differences and are very near completion.

III. Distinctively-Flavored Cigarettes

Tests of 85mm and 100mm Merit type cigarettes with WS compound vs. 85mm and 100mm Salem Lights are in the field with results expected in August. The extended smoking studies using menthol and non-menthol smokers are in the field. Various methods of application of WS compound are being investigated with the assistance of other divisions within R&D as well as Manufacturing and Engineering.

IV. Tobacco Blend Program

Subjective evaluations of burley grades have been completed. Evaluations of Oriental grades are expected to begin later this month.

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III. Services To Other Groups, PMI

The first shipment of casing flavor 7397-97 has been prepared for Brazil.

Additional samples have been made with ET from storage. Flavor evaluations are in progress on Marlboro cigarettes made with ET from two different lots.

Marlboro cigarettes with Oriental from drying experiments have shown no obvious flavor differences. Evaluation of 100% blends of the Oriental are in progress.

IV. References

1. Notebook #7396, pp. 192-200

2. Notebook #7397, pp. 137-149

3. Notebook #7564, pp. 1-8

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F. W. Smith

Louise W

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Bench scale tests using the KOH/steam method gave alkaloid reductions of 11, 25 and 36% with 1, 3 and 5 wt.% KOH added. Large scale trails were also run, varying tobacco feed rate, KOH added, VST and ADT steam flow rates, ADT air flow rate and ADT exit OV. Preliminary results have shown up to a 13% alkaloid reduction at tobacco rates of 350-400 lbs/hr (3% KOH added).

R. Z. Hall

/deb

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CHARGE NUMBER: 1716

PROJECT TITLE: Tobacco Microstructure

PROJECT LEADER: Mary Ellen Counts

PERIOD COVERED: July 1-31, 1980

DATE OF REPORT: August 1, 1980

I. TOBACCO RESEARCH

An extensive microstructural investigation was coordinated (Projects 1716 and 1901) for another "thin" vs "bodied" expansion study to examine the relationship between relative stalk position, epidermal cell size, and the "CV potential" of different grades of flue-cured bright and burley tobaccos. Generally, the largest average epidermal cell sizes were observed within the "thin" (lower stalk) grades for both bright and burley tobacco. It was anticipated from these data that the "thin" tobaccos of burley and bright would be found to have generally higher CVs than those of the "bodied" tobaccos. Cylinder volume data have not been received to date. Details of the microstructure analyses data are found in a memo of July 21, 1980 (I. L. Uydess and the Microstructure Analysis Laboratory).

A cooperative effort is in progress with Project 2106 involving heat treatment of RL. Preliminary studies on the relation between RH, OV, and CV of extracted RL will evolve into kinetic studies of RL heat treatment to determine the tobacco components contributing to cross-linking.

An initial investigation of morphological and chemical characteristics of flue-cured tobacco was completed and a special report on this subject is in preparation.

II. MISCELLANEOUS

Several requests were made for Microstructure assistance during this period. Included were:

-Assistance was given to the City of Richmond Bureau of Air Pollution with energy dispersive x-ray analysis and scanning electron microscopic examination of collected air particulates.

-Image analysis was used in a cuts per inch study evaluation with Project 1005. It was also used in determining the size distribution of charcoal material used in tobacco/charcoal separation studies (Project 1307).

-SEM, EDS, and optical microscopy were used in analyses of scrubber residues from Pilot Plant CO expansion process gas scrubbers (Project 8204).

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B. Cambridge (Project Trinity)

National POL tests have been requested for four menthol candidates of the 4 mg 100 mm Cambridge. These tests should be completed by early October 1980.

Work continues on specifications for a 1 mg 100 mm Cambridge.

C. Project Milds

Initial candidates for this program to produce a "mild" tasting cigarette with flavor were contaminated and will have to be remade.

II. INSTRUMENTATION

A. Eastman Rapid PZ Analyzer

Initial design strategy for incorporating the Eastman design for a rapid PZ analyzer into a usable test station has been formulated. Parts for this project are now beginning to be received.

B. Eastman Modified LaserMike

A technical evaluation for the Eastman modified LaserMike was written for presentation at the 1980 CORESTA meeting in Manila.

C. Combination PDI/DDI

An operating and calibrating procedures manual for the new combination pressure drop and dilution instrument was completed in July 1980.

W. G. Houck, Jr.

WGH/lad

REFERENCES

[1]POL N-4150
[2]POL N-4152
[3]POL N-4145
[4]POL N-4155, 4156, 4157
[5]HTI 5134/7084, 5135/10061, 5136/7085
[6]VP 4758
[7]POL 7044
[8]POL 7053
[9]POL 7049
[10]POL 2037, 2038, 2039

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CHARGE NUMBER:	2306
PROJECT TITLE:	Flavor Transfer
PROJECT LEADER:	R. M. Ikeda
PERIOD COVERED:	July 1-31, 1980
DATE OF REPORT:	August 7, 1980
WRITTEN BY:	R. M. Ikeda

The annual report is being written.

R. M. Ikeda

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Source: https://www.industrydocuments.ucsf.edu/docs/sjjm0000

REFERENCES

1. Memo: D. M. Teng to Distribution, July 7, 1980, "Sampling of DBC Bright and DBC Burley Lines for Microstructure Analyses."

2. Memo: D. M. Teng to Distribution, July 15, 1980, "Samples for Microstrucutre Analyses (Thin/Bodied Bright and Burley Lines)."

3. Memo: I. Uydess to Distribution, July 21, 1980, "Expansion of Thin vs Bodied Tobacco: Epidermal Cell Size and Stalk Position Analysis."

4. G. M. Nixon, P.M. Notebook #7208.

5. E. D. Mooz, P.M. Notebook #7443.

6. S. Tenhet, P.M. Notebook #7225.

/jsa

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both of these in the near future.

Visits were made to Corn Products Corp. in Argo, Illinois, to discuss starch paste, to the R & D facilities of the H. B. Fuller Co. in St. Paul, Minnesota, and to Upaco, located in Richmond.

Samples of Upaco 6624 and variations on 6624 were run on the Max S-based adhesive tester in the presence of Upaco personnel. The samples' performances were poor. Upaco intends to re-formulate.

Larry Probst and Paul Fisher of Union Chemicals visited our facilities -- both R & D and the Manufacturing Center. The visit focused on our usage of one of their hot melts in filter making.

MISCELLANEOUS STUDIES (M. Logue)

Carbowax Study

A report has been completed on the Carbowax study on the charcoal component of Parliament 100's combined filters. The following conclusions were made based on the data:

1. There was no significant correlation between the firmness of the plug at the time it came off the machine and the temperature of the Carbowax at the time of application.

2. There was no significant correlation between the final firmness of the plug and the temperature of the Carbowax at the time of application.

3. There was no significant correlation between the curing time of the plug and the temperature of the carbowax. The plugs became moderately firm in 4 to 5 hours and maximally firm in 24 hours. There was no increase in firmness at 48 hours.

Without altering the method of application of the Carbowax or some other factor (e.g., the tow) the minimum curing time of the plugs is 4 to 5 hours.

GPC

The analyses of several polymers prepared by members of Project 2500 were completed. The polymers were analyzed by GPC to determine the molecular

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CHARGE NUMBER: 2100

PROJECT TITLE: New Product Development

PROJECT LEADER: W. G. Houck, Jr.

PERIOD COVERED: July, 1980

I. NEW PRODUCTS

A. Merit Ultra Lights

Final returns (74%) from the POL test[1] of the 5 mg M + 199 vs. Merit indicate that the Merit is preferred by 0-5 mg, 5-10 mg, 10-15 mg and Merit smokers. Final returns (78%) from the POL test[2] of the 5 mg M + 199 vs. VUL indicate that VUL is preferred by 0-5 mg, 5-10 mg and Merit smokers. Final returns from the POL test[3] of the 5 mg Merit-type vs. VUL, however, indicate that the Merit-type is preferred by 10-15 mg smokers and Merit smokers. 5-10 mg smokers indicate split preference.

Currently in the field are POL tests[4] of the 4 mg Merit-type vs. Merit, 4 mg M + 199 and VUL. In HTI testing[5] are the cork and white-tipped 4 mg M + 199 vs. Merit, and the white-tipped 4 mg M + 199 vs. VUL. The cork-tipped test vs. VUL was cancelled. Topline data from these studies are expected the week of August 18, 1980. Also in the field are the identified Mall Packaging Studies. Initial data are expected by August 11, 1980.

The VP Multipack[6] which contains Merit 85, VUL, 4 mg M + 199, 5 mg M + 199 and 5 mg Merit-type is now scheduled for shipment the week of August 11, 1980. The 5 mg 100 mm M + 199 (higher RTD) is currently in the field vs. Kent III 100,[7] awaiting shipment vs. VUL[8] and being remade vs. Merit 100.[9]

Data (50% returns) from the mini-Mall Studies indicate directional preferences for the 4 mg Merit-type and Merit when tested vs. the 4 mg M + 199. The 4 mg Merit-type vs. Merit results indicate a split preference.

The 4 mg M + 199 menthol cigarettes for Mall and POL testing[10] are being held in favor of changing the menthol testing to the Merit-type blend as soon as possible.

A second factory run was completed on the M + 199 blend on August 5, 1980. An initial factory run on the Merit-type blend is scheduled for the week of August 11, 1980. As with the M + 199 cigarettes (4 mg) will be made and packaged.

Cigarette models of short-range modifications to the Merit-type blend are currently being made. Additional short-range modifications to the M blend are to follow.

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CHARGE NUMBER: 2501

PROGRAM TITLE: Nuclear and Radiochemistry of Smoke

PROGRAM LEADER: Roger A. Comes

PERIOD COVERED: July 1 - July 31, 1980

DATE OF REPORT: August 4, 1980

I. Puff Spreading[1]

Experiments are underway directed toward the potential enhancement of flavor response by the smoker while maintaining the lowest practical chemical concentration level. It has been assumed by some P.M. researchers that, if a flavor can be enriched in the early part of a puff, the subjective response to the smoker will be at its highest. The latter part of the puff could then contain less of the flavor. This assumption is being investigated by the use of a puff-spreader device. Initial experiments are being conducted to determine the individual total carbon contributions within a single puff by each individual ingredient in the 2R1 cigarette. These individual carbon contributions will then be combined to produce the total carbon smoke profile during a puff. The effect of puff number will also be determined. This total carbon profile will then be used to ratio data on individual smoke component profiles and to study these results in light of changing cigarette designs.

II. Glanded vs Glandless Tobacco[2]

Initial runs to determine the soluble ^{210}Po content of the WSC from the glanded and glandless tobaccos have been completed. The activity on a pCi/gWSC basis is within the range we have found previously for most WSC's. A 20-25% reduction in ^{210}Po activity is noted in these runs for the glandless tobacco WSC. This data will be verified by additional determinations. Future work will involve activity determinations on the filler from the cigarettes from which this WSC was derived.

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Source: https://www.industrydocuments.ucsf.edu/docs/sjjm0000

5. REFERENCES *(continued)*

7. Rapp, K. E.; Tickle, M. H. Notebook No. 7437, pp. 5-8.
8. Rapp, K. E.; Tickle, M. H. F80-8, -9, and -10: *Salmonella*/microsome activity. Memo to R. A. Pages. 1980 July 25.
9. Meyers, R. Notebook No. 7465, p. 132.
10. McCuen, R. Notebook No. 7508, p. 65.
11. Ayers, D.; Penn, J. M. Notebook No. 7551, pp. 17, 18.
12. Amacher, D. E.; Paillet, S. C.; Salsburg, D. S.; Roy, V. A. Analyses of mouse lymphoma TK assay results for statistical significance via a two sample t test. 10th Annual Meeting of the Environmental Mutagen Society, New Orleans; March 8-12, 1979; p. 82.
13. Penn, J. M. Notebook No. 7378, pp. 149-150.
14. Warfield, A. H. Determination of carbonyl compounds in cigarette smoke by reversed-phase high performance liquid chromatography. Special report 80-130. 1980 May 12.
15. Weissbecker, L. Notebook No. 7467, pp. 79-81.

R. McCuen/np

np

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Source: https://www.industrydocuments.ucsf.edu/docs/sjjm0000

CHARGE NUMBER: 2105

PROJECT TITLE: Filter Development

PROJECT LEADER: Walt Nichols

PERIOD COVERED: July, 1980

I. WRAPLESS FILTER - FILTRONA NWA

To further understand the effects of plasticizer application on the process, attempts were made to quantify plasticizer particle size. A cascade impactor was used by Physical Research personnel to approximate particle size. The Filtrona applicator was compared to the Hauni AF-2 system. Plasticizer particle mass mean diameter was about 30 µm and >50 µm, respectively. The data supports conclusions that small particles increase fiber collapse and loss of surface area. Further work is being done with the Hauni system to optimize particle size for specific tow items.

Process parameters for 3.9/40,000 tow were identified and yield was determined. This tow item will be used on conversion of Marlboro 100's to NWA. Compared to wrapped filters, a 10% increase in tow weight is required to achieve equal performance with NWA.

Tipping trials with a Mollins PA-8 were conducted at the Manufacturing Center. Ajax AC-695 was evaluated at 5000 cigts./min. Some wrinkled tipping paper was noted. Taste and odor approval was obtained. Further tipping trials will be conducted by Manufacturing Engineering. Initial filter specifications were forwarded to Engineering.

II. MENTHOL APPLICATION

Aged samples prepared with mentholated foil from Rothmans have equal menthol transfer (cigt. to cigt.) after two weeks in storage. Menthol loss from application to the pack is approximately 20%.

III. EXTRUDED TOBACCO

Sample formulations of 50% H_2O and 50% tobacco and gum have been tested for viscosity. The typical viscosity is approximately 20,000 centipoise and changes very little during prolonged mixing. Sheet material produced from these formulations typically has tensile strength > 1 kg. in the direction of calendering. The opposite direction is being effected by calendering and borders on acceptable tensile strength. Further work is being done with various mesh size tobacco dust.

W. A. Nichols

WAN/lad

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B. Cellulase Production Study

A fifteen-day time study for production of Avicel induced Tv cellulase was run. Activities in different enzyme fractions, pH change, sugar content change, and change in biomay were studied.[5,6]

C. Electrophoretic Determination of Cellulase Components

An acrylamide gel electrophoresis run was made. The fourth enzyme band which was missing in the recent enzyme preparations was detected in small quantity in the last enzyme preparation; but it was still small compared to the bands from last year's preparations. A small increase in expansion (7-10%) was noticed in cellulase treated samples over controls. The causes of different enzyme productions (the 4th band) will be investigated.[5]

D. Stress-Relaxation

A report on the stress-relaxation study of cellulase treated tobacco was prepared and will be issued shortly.[5]

E. Equipment Set-up

A Perkin Elmer HPLC and a Beckman DU-8 spectrophotometer were set-up and are ready for use.

III. DENITRATION

A. Scale-up

Preliminary meetings have been held with Chemap S.A.

Plant designs including plot maps, Philip Morris, U.S.A. Engineering specifications, tank sizes and utility requirements were discussed, and presented. In addition, production scheduling as it effects plant design was considered.

As additional information is acquired, it will be transmitted to Chemap for design inclusion. Future trips to Chemap (Woodbury L.I., U.S.A.) are anticipated and will include R&D, Process Engineering, Park-500 and Third Line personnel.

B. Additional Work

Within the next week, additional work will be performed to determine heats of reaction and the effect of denitrated liquor pH on sheet tensile strength. The work will be done at R&D and Park-500.

C. FTR Process

All equipment and supplies for FTR usage have been received. Equipment is being modified where necessary and will be ready for FTR usage on schedule. Necessary assistance will be provided to FTR personnel when needed.

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II. CO_2 Expanded ES

Rolled/cut bright stem and a 50/50 bright stem/bright strip blend were CO_2 expanded in the Phase III system at two feedstock moistures (12% and 20% OV). The reordered equilibrium CV/OV values for the expanded pure stem material were 35.9/13.9 and 42.8/13.9 for the 12% and 20% OV feed, respectively. Results for the 50/50 blends were about what would be predicted by blending the individual materials.

III. Alkaloid Recovery

Results of the initial scrubber efficiency test (fresh water in the system) showed a 100% efficiency in removing nicotine from the tower tail gas. Subsequent closed loop operation again showed the alkaloid content in the liquor levelling off at 0.5%. This has been traced to tower warmup with the normal steam atmosphere process gas. This caused recovery of tower steam, but no alkaloids since tobacco was not being processed. Steam condensate lines are being bled to also prevent dumping of condensate into the tower system during steam startup.

Cation exchange resins (hydrogen exchange type) have been ordered to allow evaluation of this technique for recovering nicotine from the scrubber water. It is planned to utilize scrubber water directly as makeup to the burley strip washer when this unit becomes operational. (In a production situation, washing ≈ 30% of the burley strip would allow direct utilization of the scrubber overflow from the manufacturing expansion facilities).

IV. Miscellaneous

Airco samples 77-80 were processed and shipped. Tests were conducted to evaluate expansion tower equipment as a means for removing alcohol from WS cut filler.

Personnel from this project and Project 1801 assisted in the production test evaluating Westab impregnated filler in the MC/DET tangential tower. A trial summary will be issued by Westab QA. Equilibrated CV's appeared to be reasonable, especially in light of the high tower exit OV's (≈ 5%) which were experienced. Tower exit OV's were higher than the values obtained in the pilot plant (≈ 3 1/2% OV) at the same temperature, which is attributable to the higher tobacco loading (4,000 lbs/hr versus an equivalent pilot rate of 1,700 lbs/hr).

R. L. Hall

/deb

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CHARGE NUMBER: 4010

PROJECT TITLE: Brand Modifications

PROJECT LEADER: W. A. Geiszler

PERIOD COVERED: July, 1980

WRITTEN BY: Jose Nepomuceno

I. BRAND MODIFICATIONS

A. FTC Tar Reductions

1. Marlboro

Cigarettes for an expanded HTI test of a 14 mg Marlboro KS versus the current 16 mg product are being remade due to tar deliveries below target. Modeling on the Marlboro LS and 100's reduced tar versions are continuing.

2. B & H Lights

Cigarettes for a POL testof a 9 mg B & H Lights menthol product are being remade due to unsatisfactory analytical results.

Several models for a 9 mg B & H Lights regular product have been tested but were all judged different from the control by the flavor panel. Several other models are in the process of being tested. The same blend as used in the 9 mg menthol product is included.

3. Merit 100's

Models of Merit 100's with a 9 mg tar delivery are awaiting analytical results. If the tar deliveries are satisfactory, subjective testing will follow.

4. Marlboro Lights

Models of Marlboro Lights 85 with 9 mg FTC tar delivery are awaiting SEF and Flavor evaluation. One model has been found to be acceptable in all aspects but harshness.

100 mm models are being evaluated in SEF. If an acceptable model is found, a POL test will be issued in August.

5. Parliament 100's

Models of Parliament Lights 100's with 9 mg FTC tar have been made and are awaiting SEF and Flavor evaluation.

6. Saratoga Lights

A new filter tow item was incorporated into additional test models of Saratoga 100's as requested by Operations Services. These models with lowered tar deliveries and reduced cigarette RTD's are presently awaiting analytical results.

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CHARGE NO: 1703

PROJECT TITLE: FILTRATION CHEMISTRY

PROJECT LEADER: C. B. Hoelzel

PERIOD COVERED: July 1-31, 1980

DATE OF REPORT: August 5, 1980

ADHESIVE PROPERTIES (T. E. Majewski)

Evaluation of headspace analysis of PVAc side seam adhesives as a possible method of correlating analytical with subjective data was started. Using National 25-1088 PVAc side seam adhesives that are being used in Louisville, 20th Street and Manufacturing Center, a G.C. method appears feasible in comparing the headspace of the adhesives. Problems in obtaining representative samples need to be worked out as well as the best G.C. parameters for analysis. Adhesives which did not pass the odor and taste tests in cigaretes will be screened using the method at hand to see if changes are apparent in the G.C. chromatogram when compared to the National 25-1088 PVAc adhesive.

ADHESIVES TECHNOLOGY (T. V. Van Auken)

Last month we found that materials used as lubricants on the PA8 tippers in Bay 3, when machined with the adhesives, cause the tipping adhesive -- Ajax 563-8 -- to congeal into a rubbery mass. These lubricants are either soaps (Chemesco hand soap ("pink hand soap") and Boraxo) or hydrocarbons (petrolatum, foil oil, and Nevastane). Through tests on the PA8-based adhesive tester we have now found that Carbowaxes which are potential lubricants for this application do not cause Ajax 563-8 to congeal into a rubbery mass. On the contrary, the Carbowaxes tested may cause Ajax 563-8 to lose viscosity when they are machined together in the PA8. This effect, if it is real, is not so harmful as congealing. The Carbowaxes which have been tested are Carbowax 600, which has two hydroxy end groups, and Carbowaxes 550 and 750, which have one hydroxy end group and one methoxy end group. Carbowax 600 is a liquid. Carbowax 750 is a solid, and Carbowax 550 has almost solidified. Manufacturing would like to have a soft solid for ease of application. Possibly this goal can be reached with a mixture of Carbowax 550 and 750, or by Carbowax 1000. We plan to examine

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constant while building up only slight pressures. Preliminary results indicate that heat treatment can increase CV even if the OV is held essentially constant. Marlboro filler has been found to exhibit signs of charring under these conditions even though the treatment temperature was only 200°F. Work is in progress to confirm and expand these findings.

Work on the effects of redrying guardited oriental tobacco to 12% OV has been started. An experimental program has been devised in conjunction with A. Kallianos. Samples have been obtained from Universal Leaf in Petersburg.

Cassandra Owen has joined the group on a temporary basis and will be assisting on this area of the project.

Peter Martin, Jr.
D. L. Bonyasy, IV

/ev

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IV. THERMAL INVESTIGATIONS

A heat-measuring device has been constructed by Engineering Services to determine cigarette heat flux. The device will be used for investigation of the relationship between static cigarette heat output, peak coal temperature and static burn rates.

B. Goodman

B. L. Goodman

BLG/lad

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chemical analysis to determine which coatings are causing or retarding flare-up.

C. Hot Melt Tipping

Various PA8 tipper experiments with tipping papers precoated with hot melt adhesives showed unsatisfactory adhesion results. Tipping with a thicker coating of hot melt glue with a lower melting point is scheduled for testing.

Jose Nepomuceno

JN/lad

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III. Greenhouse[3,4,5]

a. The sixth ^{14}C-tobacco run was started on 7/15/80 in the large plant growth chamber system in the greenhouse. Four plants of Coker 411 bright tobacco are currently being grown to replenish our supply of ^{14}C-bright tobacco for use in labelled cigarette smoke studies.

b. A literature search on hydroponics as related to tobacco culture has been completed in anticipation of future greenhouse work which may entail growing tobacco by this procedure. (Acc. # 80-201, "Recommendations for Hydroponic Culture of Tobacco at Philip Morris for Ca-45 Study" by E. A. DuRant)

In addition, calcium analyses have been obtained for greenhouse grown tobacco:

	% Calcium	
	Bright-Coker 411	Burley-Ky.10
Top Leaf	1.7%	1.1%
Stem	1.6	0.6
Middle Leaf	2.3	1.8
Stem	1.3	1.3
Bottom Leaf	3.7	4.0
Stem	1.1	1.5

The results on the bright leaf are in line with literature data. The burley data appears low when compared to field grown burley leaf (approx 8%).

c. Tobacco plant materials were provided as requested for Project 1901, Manufacturing Center Reception area, and the Electrical Engineering Services Group.

IV. Miscellaneous[6,7,8]

a. Instrument maintenance - Efforts are underway to provide five complete counting systems for the gas chromatographs for Projects 2501 and 2525. Currently three complete systems are operational.

V. References

1. R. Newman, R. W. Jenkins NB 6549
2. K. Barlow NB 7547
3. R. Bass NB 7296
4. G. Newell NB 7295
5. A. DuRant NB 7435
6. B. Francis NB 7486
7. A. Frisch NB 7309
8. G. Segura NB 7502

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The application of HPLC to work of the Chemical Research Division has begun. Already several members of the division outside our project are skilled in the use of the analytical HPLC's.

Some in depth separation development has been accomplished this month with respect to the purification of several grams of compounds 7415-89-1[5] and 7462-19 for Project 2500. Preparative separations were performed on the following samples: 7415-89-1 (9 g), 7386-36a (4 g), 7446-46 (40 g), and 7462-7 (4.5 g).[6]

REFERENCES:

1. Chavis, M.
2. Barlow, K.
3. Haut, S.
4. Edmonds, M.
5. Brady, J.
6. Core, M.

S. A. Haut

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B. Filter Length Changes

1. Virginia Slims Menthol

The Virginia Slims menthol model with the 27.5 mm filter containing 3X menthol in the filter plasticizer has achieved parity subjectively with the current product with the 25 mm filter. Preliminary specifications have been forwarded to Operations Services.

2. Marlboro Lights and Merit

POL tests for both Marlboro Lights and Merit with 27 mm filter versus the current products with 25 mm filters are awaiting subjective evaluation in POL testing.

C. CO Reduction

National POL tests have been requested comparing production Merit 85 cigarettes versus a low CO delivery Merit and a low CO delivery Merit with added flavors. These tests will be shipped upon completion of analytical, SEF and Flavor Group evaluation.

II. PERFORATION

A. Laser Perforation

Calibration experiments for the optical porosity monitor showed good correlation between pressure drop and light intensity. However, varying sensitivity functions were observed from detector to detector. A different detector configuration is being explored.

Cigarettes were made to test the feasibility of the laser ramping project. These cigarettes are currently under evaluation.

B. Parliament Lights 100

Hard mouthpiece papers to be used for the reduced tar delivery models of Parliament Lights were successfully perforated with the laser system at the Midlothian Engineering Center. Models are scheduled to be made in Manufacturing using these papers.

III. TIPPING EVALUATIONS

A. Lip Release

A SEF test involving several white tipping papers showed detectable differences between papers with and without a nitrocellulose coating. Cambridge cigarettes using nitrocellulose-coated tipping have been made and will be tested subjectively in-house.

B. Filter Flare-Up

Recent testing indicates that the cause for flare-up can be narrowed down to the type of tipping paper used. Samples have been submitted for

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PROJECT CHARGE: 1706

PROJECT TITLE: TOBACCO PHYSICS

PROJECT LEADER: D. B. Losee

PERIOD COVERED: July 1-31, 1980

DATE OF REPORT: August 8, 1980

TOBACCO DECOMPOSITION STUDIES (1,2)

EGA-GC data from experiments on J8LC bright tobacco samples in 0-50% O_2 are being analyzed.

CIGARETTE COMBUSTION/PYROLYSIS CONTROL (1,2,3)

EGA-GC combustion (22% O_2) runs have been made on α cellulose, 5% NaCl + α cellulose and $CaCl_2$ + α cellulose. Both salts lowered the total amount of CO generated.

Pyrolytic thermal weight loss data have been collected on washed Marlboro filler and washed Marlboro filler with salts (NaCl, KCl, Na_2CO_3, $CaCl_2$ and $MgCl_2$) added at the 5% level. Among the added salts Na_2CO_3 addition had the greatest effect on the weight loss seen in the "tar forming" region (increases) as well as in the "char forming" region (decreases). This salt also greatly decreases the temperature of the maximum rate of weight loss in both the "tar forming" region and the "char forming" region. In general, the monovalent chlorides slightly increase the weight loss in the "tar forming" region and decrease the loss in the "char forming" region, while the reverse is true for the divalent chlorides.

With an eye toward statistically significant coal temperatures, data on CO/puff versus coal temperature for pure MF blend components are being analyzed.

An apparatus originally designed for collecting condensate in temperature intervals (fractions) between ambient and 900°C is being redesigned. Problems associated with trapping all of the condensibles have arisen with the original design.

PHYSICAL CHARACTERIZATION OF TOBACCO (2,4,5)

A meeting was held with a Barnes Engineering representative to discuss the feasibility of measuring tobacco particle temperatures in the expansion

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B. Optimizing the Activity of Highly Active Fractions[4]

Others have reported that the response of very active samples is reduced as the microsomal concentration is increased.[6] To determine if the standard microsome concentration (35 µl/plate) employed in our laboratories was not yielding misleading results, three different volumes of microsomal mix were tested with a series of HPLC or LC purified burley WSC fractions. As expected, the lowest level of microsomes tested (15 µl/plate) produced the highest specific activities. However, the relationship of one fraction to another remained unchanged regardless of the microsomal concentration used. Thus, the active fraction(s) will probably not be missed using the standard level of microsomes.

C. Assay Modifications[7]

In an attempt to streamline the assay protocol, several experiments have been done to examine the effects of freshly dosing *versus* predosing the *Salmonella* cells. Also, we have examined the question of culture preparation as it pertains to occasionally high spontaneous backgrounds. While no firm conclusions can be made at this time (more experiments are needed), the results are similar regardless of the method used to dose the cells. The question of occasional high spontaneous backgrounds is still unanswered but does not appear to be related to how the culture was prepared from the master plates.

D. Additive and WSC Testing[7]

Three samples were tested at the request of J. L. Charles and a memo was issued concerning the results.[8]

3. L5178Y THYMIDINE KINASE MUTATION ASSAY

A. A Pure Chemical Negative Control Compound[9,10,11]

Efforts are still in progress to find a negative control compound for this system. Everything tested thus far (succinic anhydride, nicotine, ethanol, and DMSO) that we thought would be negative has been positive. Recently, methanol and anthracene have been examined. Results on the former compound are not complete while those concerning the latter agent suggest it is negative. However, the anthracene data were clouded by solubility problems with the stock solution. In order to obtain significant cell killing, a precipitate was observed in the stock solution as well as in the treated culture.

Our studies with ethanol have now been completed and as indicated above ethanol is weakly positive although other investigators have reported it to be negative.[12] It was interesting that when first

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CHARGE NUMBER: 4009

PROJECT TITLE: Smoke Studies

PROJECT LEADER: Barbro Goodman

PERIOD COVERED: July, 1980

I. HUMAN SMOKER SIMULATOR

Several product studies are nearing completion. The smoker response study of three Macho candidates is described in detail in a special report titled "Smoker Response Evaluation of Macho I, II, III Versus Marlboro 85 and Merit 85," Acc. number 80-206.

Complete particulate and gas phase data have also been collected for human smoking parameters of selected ultra-low delivery cigarettes. The smoking parameters and deliveries of two different batches of Kent III cigarettes are being compared to a Philip Morris duplication of the Kent III and to our 3 mg Elite candidate. A special report will be issued in September.

A smoker response study of 5 mg Merit Ultra Lights candidates has progressed through the profile recorder stage. Smoking profiles of the experimental cigarettes will be compared both amongst themselves and to a control Merit 85 as well as to Vantage Ultra Lights. Simulator delivery data will also be collected and compared to CI smoking data.

II. WRAPPER MODIFICATIONS

Additional runs applying three different concentrations of WS-14 to an 8-second wrapper have been made using the pipe coater. The coated wrappers are being analyzed for percent pickup versus solution concentration for determination of the desired application rate to be used on 12-second wrapper.

A time table for the base paper study has been approved. Initial investigations will involve a study of the effect of cation to anion ratio of burn additives on gas phase delivery. A non-additive base paper will be used for the coating of primarily sodium and potassium citrate. A related study has been initiated to determine the effect of wrapper porosity at low citrate levels on cigarette gas phase delivery.

III. COMPUTER APPLICATIONS

A rough draft of the BASIC conversion guide has been submitted to the computer group. The guide outlines requirements for transferring Basic programs from the Sigma to the Dec computer.

A Syntax Scanning Program is under development to aid the conversion of Basic programs. The Dec program will scan a Sigma Basic program, make some changes and flag other errors for user modification.

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Charge No.: 1503

Program Title: Modified Smoking Materials

Period Covered: July 1-31, 1980

Project Leader: G. D. Keritsis

I. Low Nitrogen Tobacco

The alkaline extraction of cured burley and bright tobacco removes over 83% of the non-nitrate nitrogen but at the expense of a 70% tobacco weight loss. Efforts are being made to recover nitrogen-free tobacco solubles for reconstitution using such techniques as acid precipitation, ultracentrifugation and ultrafiltration. Several samples were prepared by these treatments and submitted for analysis.[1]

II. Denitration of Tobacco Solubles using Electrodialysis (ED)

During the denitration runs with Park 500 tobacco solubles in the pilot plant electrodialysis unit, it was noticed that certain solid residues were accumulating in the solubles and brine manifolds adjucent to the unit's cathode plates. This problem was not evident when the laboratory unit was tested. After a thorough investigation, it is now thought that this build-up is due to the exposure of the cathode plates in the pilot unit to the tobacco solubles and brine manifolds. To study this phenomenon further the laboratory unit was modified to produce similar conditions and the results were comparable to the pilot unit.[2] Consequently, Ionics supplied new cathodes having isolated plates for a trial run beginning August 6, 1980 by project 1307 personnel.

Additional efforts are being made to improve the ED process by pretreating the tobacco solubles in order to reduce its colloidal nature which should allow a more uniform flow at higher levels of tobacco solids and NO_3-N. This will permit a more efficient rate of denitration with a smaller ED unit.

Some of these pretreatments of the tobacco solubles involve the neutralization of the colloidal charges followed by ultrafiltration or centrifugation. Laboratory studies indicate that the colloidal materials in the tobacco solubles responsible for the high viscosities are higher than 50,000 in molecular weight; and that at 35 psi, 78% (w/w/) of the solubles containing 82% of the NO_3-N pass through a 50,000 molecular weight cut off CA ultrafiltration membrane.[3] A small pilot unit equipped with 1,000 and 20,000 molecular weight cut off polysulfone UF membranes has been obtained for evaluation at higher pressure (50-100 psi).

ED systems that allow the selective removal of ions (anions or cations) from the tobacco solubles are also being studied with the use of the laboratory ED unit and existing membranes.[4]

2022179832

Source: https://www.industrydocuments.ucsf.edu/docs/sjjm0000

CHARGE NO.: 1803

PROGRAM TITLE: New Primary Processes

PERIOD COVERED: July 1-31, 1980

PROJECT LEADER: R. G. Uhl

I. Pneumatic Conveying

Evaluation of double-bladed (reinforced) fabrica tips in the rotary airlock of the vacuum convey system tangential separator was continued. The 75% reluction in air leakage across the rotary lock, as compared with the single-bladed tips, was maintained. Duct choke velocities, however, were found to be identical to those formerly obtained with the single-bladed tips indicating that rotor leakage, and subsequent turbulence in the separator, has no effect on duct conditions.

Attritions studies were conducted conveying ET at 1,500 lbs/hr over a range of air velocities. Breakage increased linearly above 2,000 ft/min. of excess velocity with both rotor types, but was almost twice as high with the high-leakage single-bladed tips. This indicates that significant air backflow through the rotary valve can be a major cause of longs fraction loss in pneumatic systems.

Tests conducted on a 14" ET conveying line at Westab showed a pipe friction factor twice as high as the value derived for the smooth-finish pilot plant duct. The higher value will be used for pneumatic system design since it agrees with handbook values for commercial pipe and is probably typical of industrial systems in normal use.

II. Alkaloid Reduction

Additional small scale testing of the NH_3/steam method was conducted on the MAG drum. Trials using higher NH_3 flows showed that there is a rapidly decreasing benefit from the higher NH_3 levels, indicating an NH_3 threshold requirement. Trials at high steam rates confirmed earlier observations of an apparent falloff, or at least a levelling, of alkaloid reduction as steam flows reached very high values. The bench tests showed that substantially less NH_3 is required to achieve the same alkaloid reduction as the steam flow rate is increased. There is some indication that steam velocity has more of an effect than total pounds of steam. Additional bench tests are planned to examine this point and to determine steam rates and residence times required to remove residual NH_3.

Large scale testing of the NH_3/ steam method in the VST/ADT dryer system showed no advantage from maximizing VST residence time (35 seconds) as opposed to running a minimum VST bed (2-5 seconds residence time). This could be expected from the MAG drum tests which showed a wide scatter in testing conducted at a residence time of 2 1/2 minutes, requiring a larger residence time (5 minutes) to show a repeatable progression of alkaloid reduction versus steam and NH_3 flow rates. Overdrying (to 3-5% OV) gave an additional 5-8% alkaloid reduction but caused a rapid falloff in reducing sugar content. Other trials showed that the use of steam in the ADT dryer is essential to achieving any reasonable alkaloid reduction.

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CHARGE NUMBER:	6906
PROJECT TITLE:	BIOLOGICAL EFFECTS OF SMOKE
PERIOD COVERED:	July 1-31, 1980
PROJECT LEADER:	R. A. Pages
WRITTEN BY:	R. McCuen
DATE OF REPORT:	August 6, 1980

1. YEAST MITOTIC GENE CONVERSION ASSAY[1]

A. Whole Smoke (WS) and Gas Phase (GP) Activity (with 6908)

The observation that burley WS collected in DMSO was more active than conventional dry impaction trapped (IT) burley WSC was confirmed and extended in the experiments conducted this month. When GP from burley cigarettes was recombined with dry IT WSC, the activity of the recombined sample was similar to that of burley WS collected in DMSO. [GP from burley cigarettes was previously shown to be inactive in this system.[2]]

The dry IT WSC, GP, and WS as well as a recombined sample were obtained from a low tar filler cigarette (LTF-IIA). When tested, the activities were similar to that obtained with the burley cigarette. That is, WS > recombined sample > dry IT WSC. Unlike the GP from burley, that of the low tar filler cigarette was active. These results are encouraging because they show that different collection procedures are trapping different and/or more active agents.

B. Cell Stock Sensitivity

Recent experiments involving another flask of the same batch of cells we had been using in this assay showed this new flask of cells to be extremely sensitive (more active) to WSCs. The spontaneous conversion frequency and the response to a pure chemical were similar regardless of which flask of cells was used. Only the response to WSCs were different, however, the relationship between two WSCs remained the same (*e. g.*, IM > 2R1). If we could determine what factor(s) is responsible for the increased sensitivity of the cells in this flask, we would have made an important contribution to understanding how this assay works. One problem experienced by ourselves and others[3] is the low response of most agents when tested in this system.

2. *SALMONELLA*/MICROSOME ASSAY

A. Pyrolysis (with 6908)[4]

As reported previously,[5] the pyrolyzate from 2R1 filler pyrolyzed at 200 and 300°C was not active. These studies were extended at pyrolysis temperatures of 350, 400, and 500°C. Increasing activities (TA98 with microsomes) were observed as the temperature was increased.

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PROJECT CHARGE: 2106

PROJECT TITLE: APPLIED TECHNOLOGY

PROJECT LEADER: Peter Martin

WRITTEN BY: Peter Martin and Joe Banyasz

PERIOD COVERED: July 1-31, 1980

DATE OF REPORT: August 8, 1980

LASER PERFORATION OF TIPPING PAPER

Work has continued on new product development using the original perforation system at Midlothian Complex. Some problems were experienced with the L & M Lark brand dilution being too high. This was traced to a change in the plug wrap porosity. As a large quantity of plug wrap is in storage, a new pressure drop specification was established to maintain correct dilution levels.

Calibration curves have been run on the Optical Porosity Monitor (OPM). A new diode has been ordered to improve the OPM's operation.

Bacterial growth has been found in the water cooling systems of the laser systems. This forced the use of higher water pressure to maintain the flow rates and these higher pressures caused several of the hoses in the cooling line to rupture. In conjunction with Dr. Semp, the bacterial growth was traced to city water makeup. He has recommended ways of cleaning the lines and a cooling system redesign to prevent this problem in the future. Both of these are currently under way.

The second training course on laser system operation for ET's at 20th Street is now being given. Assistance has been given on the Laser Analyst job definition and the subsequent search for suitable applicants.

ON-LINE OV MONITOR

Park 500 are evaluating possible techniques and vendors for an on-line RL monitor with feedback control of OV in the sheet. We have attended the presentations and analyzed the data presented to determine how well it could be applied in practice. The technique presented by Measurex looks promising but still seems to have no way of determining the level of the solubles.

HEAT TREATMENT OF TOBACCO MATERIALS

Experiments on heat treating tobacco at constant moisture content have been started using the pressurized containers. Experiments at 130 and 200°F indicate that the devices do indeed maintain the moisture content of tobacco

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CHARGE NO.: 1801

PROGRAM TITLE: Tobacco Processing

PERIOD COVERED: July 1-31, 1980

PROJECT LEADER: F. V. Utsch

I. Reordering of MC DET (D. Fox)

A test in the MC to confirm earlier results which indicated improved CV with longer reordering times was not conclusive; some evidence of differences in performance between the two reordering cylinders was again observed. The possibility of additional analytical work to monitor each cylinder's performance has been discussed with the DIET QA lab, and a test is scheduled to evaluate each cylinder at various throughputs. Another test with both cylinders at longer residence times on a split-flow basis is also being planned for August. The installation of thermocouples at the tower exit and across the reordering cylinders to define temperature profiles has been completed.

In the pilot plant, hydraulic nozzles are being investigated for use in reordering. Plans are for a hydraulic nozzle assembly to be installed in the 8" tower reordering cylinder to compare with air atomized water sprays. Other types of spray devices, including some relatively recent developments, are also being studied. Further studies on the effects of spray rate, temperature, and droplet size are also in progress in the Ball Impregnator.

II. Expansion Tower Design Studies

A. Gas/Particle contact (High Speed Photography) (B. Donenfeld)

Observation and filming of the 3" x 10" plastic rectangular ribbon tower has been delayed until additional parts arrive; a venturi throat section and outside wall deflector elbow section are being shipped this week. Two differing radii of curvature elbows and an offset-bulge vertical section have also gone out for bid.

At a series of tower studies meetings held this month, the decision was made to install an operational rectangular cross-section expansion tower.

B. Expansion Studies - 4" Tower (M. Mobrem)

Results from DIET expansion on the 4-inch short horizontal tower indicated that product exit OV's were higher at comparable CV's compared to the two-elbow configuration (4.5% exit OV vs. 2.5% exit OV, respectively to achieve an 80 CV). Elimination of elbows, and hence, reduction in the particle residence time was the major contributor to the above finding. However, the DIET equilibrated CV/OV curve was in general comparable to the results of the two-elbow tower configuration. The above comparison was made at impregnation conditions of P = 600 and 800 PSIG, feed OV = 12% and 20%. The statistical evaluation of DIET impregnation conditions did not indicate any significant changes in the product equilibrated CV/OV results from any of the above conditions.

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This methanol eluate was also analyzed by gc/eims (glass column packed with 3% SP-2250 DB).[2] The chromatogram contained several well defined peaks. Harman and norharman were again observed as major chromatographable components. Further analysis of ms of fraction components and comparison of gc results to capillary gc results is planned.

Considerable time was devoted to the development of new HPLC methods for separation of individual components in complex base fractions, again the methanol eluate from alumina chromatography of X6D3IM base fraction was used.[5] Ion pair systems were evaluated (sodium hexanesulfonate) but the base fraction components such as 2-amino-α-carboline appear to be too hydrophobic for this approach. Next, a number of ion suppression systems were evaluated. Using ammonium carbonate for ion suppression, a three component organic solvent mixture, gradient conditions, and elevated temperature (60°) allowed optimal resolution of any selected component in this complex mixture.

C. FLUORESCENCE DETECTOR EVALUATION[3,5]

A Perkin Elmer 650-10S fluorescence spectrophotometer was evaluated this month. Good excitation and emission spectra were obtained using the standard cell for PAHs, heteroaromatics, and 2-amino-α-carboline. The instrument was used with the HPLC flow cell in an attempt to locate 2-amino-α-carboline in the methanol eluate (alumina) of the X6D3IM base fraction. Using optimal excitation (325 nm) and emission (367 nm) the peak for the 2-amino-α-carboline was easily located in this fraction at the appropriate retention time. No significant peak could be observed using the variable wavelength UV detector with this same fraction.

Comparison of the results from capillary gc, packed column gc/eims, HPLC with variable wavelength UV, and HPLC with fluorescence detection provided strong support for the analytical capability of fluorescence detection for trace components in the base fractions.

D. BASE FRACTION AND THE *SALMONELLA* ASSAY (with 6906)

A study of WSC from LTF-IIA + proline (X6D9FDB) was initiated.[6] This is a major amino acid of bright filler and was known to produce a high activity condensate when added to an LTF-IIA matrix.[7] The WSC was fractionated to acid-neutral and base fractions. As expected, the base fraction had high activity (42 rev/μg) and a high accountability (71% of WSC activity). The bases were further separated by chromatography on ODS-silica and these fractions are being tested.

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CHARGE NUMBER: 1708

PROJECT TITLE: Physical and Chemical Properties of Tobacco

PROJECT LEADER: B. C. LaRoy

PERIOD COVERED: July 1-31, 1980

DATE OF REPORT: August 11, 1980

I. MECHANICAL PROPERTIES OF FILLER (D. A. Full)

A. Instron Simulation of Maker Compression Cycle

Experiments in this area have been postponed by renovations in Room T-427, where special humidity and temperature control equipment is being installed.

B. Coal Strength Measurements Using 100 Hz Sinusoidal Vibration

Two samples of Marlboro type cigarettes having differences in blend components were compared by making 80 individual coal strength measurements on 20 weight-selected cigarettes of each sample, provided by Dr. Lowitz. The sample, designated 'AA Tipped' and 'BB Threshed', were found to have resolvably different coal strength distributions. Modelling them by log-Guassians, the former had a slightly higher mean coal strength (i.e., it took a slightly greater peak acceleration, on the average, to shake the coal off) and a slightly smaller standard deviation (i.e., the required accelerations were not so widely scattered). Based on the modelled parameters, the coal failure rates expected for 30 g's were calculated to be roughtly 0.03% and 0.3%, respectively.

II. TOBACCO LEAF CURLING[1] (J. C. Crump, III and L. Trentham)

As reported in May, a study has been underway to investigate the effect of moisture and heating temperature on the degree of curling of tobacco when it is processed through the Proctor & Swartz dryers at the Richmond and Louisville Stemmerys. The study was initiated to provide assistance to Mr. Karl Jockel of Louisville and Mr. Frank Lipski of Richmond. Their primary concern involves the overall reduction in size, due to curling or shrinkage, of tobacco leaf when subjected to high moisture and temperatures in the Proctor & Swartz Unit after the removal of stems. This shrinkage has the result of increasing the percentage of leaf which falls into smaller sieve sizes. The purpose of the study is to obtain information regarding optimum conditions of temperature and moisture for minimum curling or shrinkage.

The results tabulated in Table I show a definite trend toward more curling of the tobacco at the higher moisture levels. Based on these results, it appears that the optimum conditions of temperature and mositure for minimizing curling are moisture levels below 16.7% OV and temperatures either in the 125°F range or between 200 and 225°F.

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CHARGE NUMBER: 1901

PROJECT TITLE: Biochemical Modification of Tobacco

PERIOD COVERED: July 1-31, 1980

WRITTEN BY: D. M. Teng and B. A. Semp

DATE OF REPORT: August 8, 1980

I. TOBACCO MATERIALS STUDY

A. Large Scale Thin/Bodied Tobacco Expansion

Assistance was provided in sampling forty-eight Burley samples and fifty-two Bright samples for thin/bodied tobacco expansion conducted at the Manufacturing Center. Sixteen of the Burley samples and eighteen Bright samples were processed and expanded. They were submitted to the Microstructure Facility for cell size determination.[1,2] The largest averaged epidermal cell sizes were observed to occur within the "thin" grades of tobacco (both Bright and Burley).[3]

B. Ratooned Tobacco

The third-year study of ratooning bright tobacco was started in the early part of the month of July. Tobacco plants were topped to 6-8 inches from the ground. A main sucker growth after the topping was saved from each plant.[4]

C. Small Scale Aging Study

The microbial profiles of the 18th-month samples from a small-scale aging study were determined.[4]

D. Tobacco Protease

Protease activities in green tobacco extract, frozen and freeze dried samples were determined. All activities were low. Work on isolation of possible protease inhibitors was begun.[4]

II. CELLULASE

A. Reducing Sugar Formation in Cellulase Treated Tobacco

Sterile cut filler was treated with water, buffer or cellulase and the reducing sugar contents were determined. Cellulase-treated tobacco showed an increase in total reducing sugars as compared with the buffer or water-treated samples (5.7% to 1.7% and 2.0%). From these results it is apparent that sugar is produced in tobacco samples due to cellulase treatment.[5]

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CHARGE NO.: 1804

PROGRAM TITLE: Expanded Tobacco - Process Improvement

PERIOD COVERED: July 1-31, 1980

PROJECT LEADER: R. G. Uhl

I. Breakage

Pilot plant trials evaluated the MC clumpbreaker rotational speed (60 rpm) against normal pilot conditions (17 rpm) and showed a small advantage {Δ% (longs + mediums) = + 1 1/2} at the lower speed. Plans are being formulated to evaluate reduced rpm at the MC. A new gearbox is being installed on the pilot clumpbreaker to investigate even lower speeds. Removal of static breaker bars at the MC does not appear feasible because of a potential problem which could result from still-frozen clumps exiting the expansion tower. (cigarette wet spots in make/pack). Some means for removing these clumps would be required before trials could be conducted in this area.

The testing of modified impregnation cylces continued in an attempt to reduce hardness and prevalence of clumps exiting the impregnator. Reducing the pilot batch size by 30-50% has shown a marked improvement. A test grid has been initiated varying feed OV, impregnation pressure and batch size (each at several tower exit OV's) to determine the overall effect on batch compression, clumping, breakage and CV. Initial results indicate that intermediate moistures and pressures (16% OV, 550 psi) minimize the occurence of a clump at one particular spot in the batch and give a more uniform impregnation.

The existing MC impregnators are rated for 480 psi which would essentially limit us to present process conditions with this equipment. The best results in this case have been obtained with a three minute gas purge following the drain cycle. However, results have been enatic with a considerable variation in the amount of improvement achieved. The best apparent process conditions resulting from this study will be used to impregnate batches which we will attempt to process with the clumbreaker removed.

Examination of the hardest clumps has shown a definite orientation of tobacco shreds, apparently in what would be the horizontal plane in the impregnator. The clumps are impervious to a compressive force applied perpendicular to this plane. However, they break up easily when a compressive force is applied parallel to this plane, indicating that some sort of "side squeezing" mechanism might be employed. This shred orientation was not apparent in reduced-size batches processed at intermediate moisture and pressure. These were randomly oriented, less compressed during impregnation and loosely agglomerated. Tests are planned to evaluate extended liquid fill/drain times to determine whether rapid fill could be fluidizing the tobacco batch and allowing it to settle in horizontal layers.

Orientation of tobacco shreds and compression of the batch during impregnation appear to correlate directly with the presence and hardness of clumps.

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CHARGE NUMBER: 2305

PROGRAM TITLE: Flavor Development

PROJECT LEADER: J. W. Swain

PERIOD COVERED: July 1-31, 1980

DATE OF REPORT: August 5, 1980

WRITTEN BY: J. W. Swain and L. Wu

I. RL Flavor

Additional RL (#605) has been made in the Pilot Plant with a reduced level of ammonia precursors. V-3818 will test the modified RL replacing RL-150B and ES in a Marlboro blend.

Two blend levels of denitrated RLs from electrodialysis and anaerobic fermentation processes are currently being evaluated with and without 150B flavors added to CEL. Preliminary evaluations have shown no significant flavor differences between the crystallized control and electrodialyzed test RLs (both paired with and without 150B) at the 19% level, but both comparisons were significantly different at the 27.8% blend level.

The same comparisons between crystallized control and test (denitrated by anaerobic fermentation) RLs showed that neither blend level was subjectively different when both RLs were flavored (150B); however, results of comparisons of these without 150B flavors showed significant differences. Further evaluations are in progress.

II. Reaction Flavors

Virginia Panel 6579 evaluating a79-3 B&H control vs. a79-3 B&H with 563RL has been completed. The test cigarette with the reaction flavored RL was found to have a good taste, to be satisfying and to be preferred by the 100mm full flavor smokers other than B&H smokers and the combined panel.

The Merit Virginia Panels have been released with 4753 in the field and 4754 awaiting shipment.

A program was initiated to study the subjectives of blending 150B RL with Reaction Flavored RL's in the 81-1 Marlboro configuration. Four blend levels of two different reaction flavored RL's are being screened.

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Source: https://www.industrydocuments.ucsf.edu/docs/sjjm0000

Charge No.: 1307

Program Title: Reconstituted Tobacco Development

Period Covered: June 30-August 11

Project Leader: G. Gellatly

I. RL Process

The third meeting of the steering committee for Park-500 Line III Design was held on 7/30/80 to make a recommendation for the design of the blending and wet slurry transfer system.[1] Various designs suggested by Sirrine Engineering Company were considered and a recommendation was made to build a new blending area to service four lines with space for additional equipment to handle a separate blend for Europe if necessary on Line III. Building a new blending facility minimizes plant downtime (1-2 weeks) and could make use of existing equipment in the present blending area for possible separate European blend processing. To modify the present blending area would entail plant downtime of six months which would eliminate our RL inventory.

Extract liquor clean-up trials were run in the pilot plant and Park-500 by Alfa Laval, the centrifuge supplier. They demonstrated that their equipment could reduce the suspended solids content of the evaporator feed liquor by 50% of that remaining in the present liquor after the Bird centrifuge clean-up. There may be insufficient time for a production trial, however, before the order for Line III centrifuges is placed.

II. RL Product

Engineering modifications to the paper machine were initiated to enable us to make larger rolls of base web. This will facilitate our running longer trials to compare the filling power of RL finished sheet prepared by Yankee drying versus tunnel drying the basic web before sizing. If these trials confirm a ten percent filling power advantage for tunnel drying over Yankee drying, a recommendation will be made to replace the Yankee drier with a tunnel drier in the pilot plant for continuous demonstration.

The breakage of RL product (as cut filler) was found to decrease when the tensile strength of the sheet increased. RL products prepared at Park-500 (one sample at 0.6 kg/in and a special test sheet at 1.2 kg/in tensile strength) and the pilot plant sheet (2.0 kg/in tensile strength) were used in the evaluation using 100% RL filler and 28% RL in MF blend cigarettes. The 100% RL filler was sieved before and after ballmilling with rubber stoppers and the longs fraction breakage was found to decrease by 10% for each unit increase of tensile strength. Cigarettes containing 28% RL will also be evaluated for firmness, coal strength and loose ends. No significant difference between filler breakage of Park-500 RL (G) and RL (TEG) was seen by sieve analysis of filler product before and after ballmilling or processing through a cigarette maker.

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Further preparative fractionation of various active "strippings" fractions from LH-20 fractions continues.[6] Samples are being submitted both for activity testing and gc/eims profiling. A number of components are being observed in these fractions by gc/eims.[2] For example, a MW 197 component (possibly a methyl amino carboline), MW 183, MW 195, and MW 209 components were located. Additional ms interpretation is in progress.

E. PRECURSORS OF WSC ACTIVITY

Equipment was obtained and set up for preparation of LTF sheet materials.[5] With assistance from Mr. G. Keritsis and Mr. J. Leik, a high organic formulation was prepared, cast, and cut.[8] This nitrogen-free filler has been submitted for analysis, cigarettes have been prepared and smoked, and the IT WSC is being tested. This material incorporates a high level of microcrystalline cellulose (Avicel-PH-101, 50 micron, FMC) instead of α-cellulose.[9] This material is being prepared in support of several ongoing precursor studies in Projects 6908 and 6906.

F. REFERENCES

1. Baker, P. G. Notebook No. 7545, p. 6.
2. McKay, C. Notebook No. 7477, p. 59.
   Kinser, R. Notebook No. 7476, p. 48.
3. Millham, J. Notebook No. 7139, p. 183.
4. Levins, R. Notebook No. 7351, p. 121.
5. Warfield, A. Notebook No. 7434, pp. 94-96.
6. Tafur, S. Notebook No. 7423, pp. 170, 171.
7. Ferguson, R. Notebook No. 7372, pp. 33, 34.
8. Petri, D. Notebook No. 7331, p. 156.
9. Gager, R. Notebook No. 7375, pp. 83, 84.

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CHARGE NUMBER: 2500

PROGRAM TITLE: Synthesis of Tobacco Additives

PERIOD COVERED: July 1 - 31, 1980

PROJECT LEADER: J. I. Seeman

DATE OF REPORT: August 8, 1980

I. FLAVOR RELEASE CHEMISTRY

The preparation of β-methylvaleroyl esters of sucrose by two different methods has now been completed[1], and preliminary pyrolysis results indicate that significant yields of β-methylvaleric acid are formed.[2] The modes of preparation for this material were the reaction of sucrose (1) with the corresponding acid and carbonyldiimidazole and (2) with N-β-methylvaleroylimidazolide. These materials are very similar to samples provided by Flavor Development by spectroscopic analysis. Excellent results are now being obtained in even simpler modes of preparing sugar esters, i.e., by reacting sucrose directly with the related acid chloride.[1]

2-Methylbutenyl ethyl 2-butylmalonate has now been prepared from the reaction of the lithium salt of 2-hydroxy-2-methylbutene and the acid chloride of ethyl hydrogen sec-butylmalonate. This important monomer will serve as an ester release polymer building block.[3]

The dianion of n-valeric acid was reacted with 3-pyridinecarboxaldehyde to give the β-hydroxy acid, 2-(2-butyl)-3-hydroxy-3-(3-pyridyl)propionic acid. This material was prepared as a potential aldehyde:acid release agent.[3] The related ethyl ester was prepared by reacting ethyl valerate with 3-pyridinecarboxaldehyde; unfortunately, pyrolysis of this hydroxyester resulted in only low yields of the aldehyde and ester.[4]

Separate pyrolyses of the two isomers of ethyl 2-propyl-3-hydroxy-3-phenylbutanoate was carried out at 200°C. It was found that one of these was ca. three times more reactive than the other. Work is currently in porgress to determine unambiguously the stereochemistry of the two isomers.[4]

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CHARGE NUMBER:	6908
PROGRAM TITLE:	SMOKE CONDENSATE STUDIES
PERIOD COVERED:	July 1-31, 1980
PROJECT LEADER:	R. N. Ferguson
DATE OF REPORT:	August 6, 1980

A. NITROSAMINES

The current trapping procedure for tobacco specific nitrosamines, 250 ml buffer in first trap, 100 ml buffer in second trap, and a TPM pad backup is being investigated.[1] The model cigarettes 2R1 and a nitrate added bright X6D3NX were weight and RTD selected for this study (2R1 1.173 g ± 1 mg 3.4" H_2O ± 0.1"; X6D3NX 1.139 g ± 3 mg 3.1" H_2O ± 0.1"). These cigarettes are being used for seven replicate smokings, processing, and will then be analyzed (gc-tea). This will provide a data base on trapping patterns and method reproducibility. The X6D3NX samples are being smoked with and without filters to also evaluate this important parameter.

A reference sample of N-Nitrosoanatabine (NAtB) was examined by probe eims.[2] The mass spectrum was in agreement with the literature for NAtB although a minor impurity was present. A glass column packed with 3% SP-2250, as used in our gc-tea analysis, was used for gc/eims of NAtB. A single component eluted, but the spectra obtained were no longer consistent with its identification as NAtB. The molecular ion now appeared at m/z 156 and the ms is consistent with identification as bipyridyl. While we cannot yet explain this conversion, it may relate to the low signal intensity observed for NAtB in the gc-tea system.

B. CHROMATOGRAPHY

The current procedure for quinoline was compared to chromatography of a base fraction on alumina. It appears that quinoline will elute with ether and harman with methanol. This procedure may be more straightforward than the present method.[3]

A sample of the methanol eluate (alumina column) from X6D3IM bases was used for capillary gc (HP 25 m x 0.2 mm SP-2100).[4] Approximately 200 peaks were detected, but peak intensity indicates that a considerable portion of the injected material did not chromatograph. Harman and norharman were identified by retention time as major fraction components. A synthetic sample of 2-amino-α-carboline eluted as a symmetrical peak under these conditions but no significant peak with the appropriate retention time was observed in the methanol eluate fraction.

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their structure-activity relationships.[12]

Nitroethylene has now been found to be an excellent Michael acceptor, having reacted to give in reasonable yield 6-nitroethyl-7,8-dihydro-5(6)-quinolone, an intermediate to the 2,2'-bridged nicotinoids. This procedure will allow us to convert 3-acetylpyridines to the corresponding nicotine compound. Racemazation studies continue on our asymmetric synthesis of 5-methylnicotine.[11]

The iodomethylations of four 1-methyl-2-(2-alkylphenyl)-pyrrolidines were carried out as well as of a number of other nicotine analogues including 2'-methylnicotine. Standard curves were also obtained on eight compounds.[14]

IV. CUSTOM SYNTHESIS

A mixture containing the tobacco beetle pheromone has now been prepared, based on detailed mass spectral evaluations. This mixture was obtained in seven steps from diethyl ketone. A major effort in purification has begun. It is hoped that individual isomeric entities can be given to A. Manzelli and S. Long such that the stereochemical structure of the pheromone can be ascertained. Large scale preparation of this material is in progress.[10]

V. REFERENCES

1. G. Chan, 7462
2. We thank L. Brown for these results.
3. H. Grubbs, 7361
4. Y. Houminer, 7424
5. F. DeBardeleben, 7310
6. C. Storms, 7412
7. We thank M. Logue for these GPC analyses.
8. R. Southwick, 7446
9. W. B. Edwards, III, 7415
10. D. Williams, 7448
11. C. Chavdarian, 7391
12. H. Secor, 7386
13. With V. DeNoble and L. Carron
14. D. DeNagel, 7472

Jeffrey Seeman

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examined, ethanol was negative[13] while more recent experiments have shown it to be positive. This discrepancy was resolved when it was found that two different microsomal preparations were used in the experiments just described. In work just completed, it was conclusively shown that one microsomal prep was capable of activating ethanol while the other was not. While the *in vivo* activity of ethanol is questionable, the *in vitro* studies suggest that its primary metabolite (acetaldehyde) is responsible for its activity. Studies are underway to determine if acetaldehyde is active in this assay. If successful, these studies would complement those done in *E. coli* concerning the activity of aldehydes in that system.[14]

4. BHK CELL TRANSFORMATION[15]

A collaborative study with Dr. J. A. Styles of ICI in England was completed this month. A cell clone isolated in our laboratories yielded a high spontaneous transformation frequency when tested in the ICI protocol using their media, serum *etc.* Using ICI's cells and their protocol with several different types of sera produced some differences and some similarities depending on what parameter was measured. For example, the cloning efficiencies and transformation frequencies were similar while the LD_{50s} were quite different. The type of sera used contributes to the variation observed in this assay. We are attempting to obtain a contaminant-free flask of ICI's cells, so that more studies can be done with this system.

5. REFERENCES

1. Thompson, L. H. Notebook No. 7557, pp. 7-12.
2. Pages, R. A. 6906 Monthly progress report. Monthly Progress Reports 80-180. 1980 July 15.
3. Thompson, L. H.; McCuen, R. W. Trip Report: The 11th annual meeting of the Environmental Mutagen Society (EMS), Nashville, Tennessee, March 16-19, 1980. Memo to R. A. Pages. 1980 April 1.
4. Drew, S.; McCoy, W. R. Notebook No. 7484, pp. 105-108.
5. Ferguson, R. N. 6908 Monthly progress report. Monthly Progress Reports 80-180. 1980 July 15.
6. Ishii, K.; Yamazoe, Y.; Kamataki, T.; Kato, R. Metabolic activation of mutagenic tryptophan pyrolysis products by rat liver microsomes. Cancer Research. 40:2596-2600. 1980 July.

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Using a literature procedure, *l*-tartatic acid was converted to *l*-bis-O-carboxytartaric anhydride, a rather remarkable organic molecule having the unusual empirical formula $C_6H_2O_8$. Perhaps not unsurprising, it may find utility for a dry powder soft drink carbonating ingredient. Samples will soon be forwarded to Dr. Will for evaluation.[3]

Efforts to prepare 2-(2-butyl)-3-hydroxy-3-(3-pyridyl)-butanoic acid in large scale were somewhat thwarted by unusual experimental problems but work is underway to overcome these. However, the related ethyl 2-(2-butyl)-3-hydroxy-3-phenyl-butanoic acid was prepared again and added to the CR file.[5] Additional quantities of sodium menthyl itaconate have been prepared for flavor development evaluations.[3]

Benzyl and β-phenylethyl itaconate monomers were synthesized and subsequently independently polymerized.[6] GPC analysis indicates that they are 90% polymer/10% monomer each.[7] Various procedures have been tried to make the related phenol and dimethyl allyl alcohol esters without success, though some promise comes from the reaction of lithium phenoxide (2 equiv) with itaconyl chloride.[6]

Successful formation of the vinyl carbonate monomer of an unsaturated ester release system has been completed. This material has also been polymerized, giving the first example of a release agent for this class of flavorant.[6]

II. FLAVOR CHEMISTRY

A total of 15 g of 12-(Z)-abienol, an important terpenoid precursor to a wide range of labdanoid flavorants, has been obtained. A portion of abienol was photooxygenated and the crude product isolated by column chromatography. The mixture of oxygenated terpenoids has a "delightful dusty-wood aroma"; it is hoped that we will be able to separate out materials known to be important tobacco flavorants. The remaining abienol was treated with m-chloroperbenzoic acid to give, following HPLC purification, a mixture of two major constituents. This is another effort to prepare important oxygenated terpenoids.[8]

Small quantities of isopropylpyrazine and *t*-butylpyrazine have been synthesized. A practical synthesis of 2-chloro-3-methylpyrazine was also developed. This latter compound should serve as the key intermediate in the preparation of 2,3-dialkylated pyrazines.[8] These will form a series of compounds to be used in a multidimensional scaling experiment with Dan Ennis from which it is hoped to correlate subjective properties pyrazine flavorants with their structure and chemical reactivity.

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However, there was an indication that 20% feed OV product yielded essentially no net breakage. (Product was hard fed to the tower, no clump breaker was involved.) This could not be compared to the results from the two elbow tower configuration because acceptable product was not obtained at 20% feed OV.

Results from Westab impregnated material indicated an improvement in the equilibrated CV/OV levels (CV > 100 at OV = 11.0%). Alkaloid and glycerine losses were comparable to the 8" tower.

The 1980 EQ CV/OV regression analysis that was conducted for ET has been completed. (Memo from M. Mobrem to H. Merritt July 15, 1980.) Plans are in progress to switch from the 4" ϕ short horizontal tower to the 6" ϕ long horizontal. This will enable us to test expansion at gas velocities of 60-70 ft/sec. at comparable lb. gas/lb. tobacco as run in the 4" diameter tower.

III. Carbamate Powder ET (F. Utsch)

Shakedown of the prototype system is in progress and the first series of tests is scheduled for next month.

IV. New Tobacco Treatments

A. Freeze Drying of Tobacco (B. Donenfeld)

The eight-batch turger conditioning run in which the excess decanted liquid was recycled into the next sample has been completed. Analytical data on the decanted liquid showed that the amounts of total solids, total alkaloids, reducing sugars and chlorides approach a steady state plateau by the eighth batch. The tobacco samples had equilibrated CV's between 53 and 65 at OV's between 11 and 13 percent. The last four tobacco samples felt sticky after they had been freeze dried and equilibrated. Complete analytical and microscopy data on the tobacco has not been received.

B. Clumping in the DIET Impregnation (R. Burde, P. Aument)

Modifications to the Phase II unit have been made to allow study of the effects of the vent direction and gaseous sweeping of the tobacco bed. The Phase II unit is not suitable to test the effect of gas sweep on large tobacco beds; thus its data is not translatable to larger units. Tests on this unit shall be concerned with the kinetics of clump formation.

A portable ultrasonic horn has been ordered for tests on breaking up clumps. An ultrasonic cavitation unit capable of working in liquid CO_2 under pressure to strip excess liquid off tobacco has not been located.

A small unit capable of impaling tobacco clump on spikes has been constructed. Preliminary observations indicate that impaled clumps break along natural cleavage planes. These cleavage planes contain less interlocking tobacco particles, split easily into small "flakes" and indicate less mechanical breakage of the total filler.

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Thuja leaf oil, comprising ca. 50% thujone, was oxidized with permanganate to give a trisubstituted, poly oxygenated cyclopropane derivative which was treated with water to form 3-isopropyl-6-ketohept-2-enoic acid. Solanone was obtained in initial experiments in one step from the reaction of 2-isopropyl-5-ketohexanal and an allylic Wittig reagent. Efforts to purify solanone are underway.[9]

A number of ketoacids were added to the CR file, and the related invention record for the preparation (process) of such compounds has been submitted.[10]

After much effort, 2-isopropylbutanal, a key compound in the preparation of numerous tobacco identical terpenoid flavorants, has been obtained. The successful route involved the ethylation of the *t*-butylimine of isovaleraldehyde. Work is in progress to improve the low yield.[10]

III. ALKALOID CHEMISTRY

With the assistance of Steve Haut, the successful use of HPLC for the purification of nicotine analogue reaction products has been successful. This is an important accomplishment, for it will allow us to prepare many nicotinoids heretofore unavailable. Thus, cis and trans-3'-methylnicotine have been obtained in sufficient quantities for testing. Several purifications of 2-, 4- and 6-methylnicotine having all three products in the total mixture were accomplished by HPLC and optically active 2-methylnicotine and 6-methylnicotine have now been obtained for the first time. The rotations were low in some cases, high in others, indicating that racemization may be occuring during the reaction. Work continues along these lines.[11,12]

6-Methylnicotine was reacted with LDA/THF and gaseous formaldehyde to give in 43% yield a hydroxyethylnicotine. This compound is the first heteroatom substituted alkylnicotine and is to be used to prepare a nicotine analogue which will react irreversibly and covalently at the nicotine receptor. Should this approach succeed, a major step toward isolating and identifying the nicotine receptor(s) will have been made. A side product in the above reaction may be 6-hydroxymethylnicotine, a very interesting compound in its own right.[11]

Additional quantities of 4-isonicotine and a number of quaternary slats have been prepared. Also, efforts to prepare a tetradeutero analogue of 3,3'-isonicotine are meeting with success. The latter compound will be useful in analyzing the alkylation of this interesting active material.[12]

We have most recently found[13] that 2'-methylnicotine is a most unusual nicotinoid. Efforts are underway to prepare additional 2'-substituted nicotine analogues in order to evaluate

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CHARGE NUMBER: 2525

PROJECT TITLE: Chemistry & Isolation of Tobacco Constituents

PERIOD COVERED: July 1 - 31, 1980

PROJECT LEADER: S. A. Haut

DATE OF REPORT: August 4, 1980

A systematic calibration of the Berthold HPLC radiochemical detector is now underway. Although this detector can be used with any form of HPLC it has been connected to our HPGPC so that the columns may be calibrated at the same time. The flow rate in preliminary data is the most influencial factor in the radiochemical sensitivity of this instrument. ^{14}C-toluene and ^{14}C-dotriacontane have been used so far as reference compounds.

Our participation with Project 2501 in the Polonium study continues.[2]

The WS-14 synthesis work has begun to show some definite trends. The preparation of the Grignard Reagent (ℓ-menthyl magnesium chloride) is now quite dependable and can be formed in essentially quantitative yield in refluxing THF. Reaction of t-butyl isocyanate with this reagent at low temperature (-78°C) tends to give a higher ratio of WS-14 to neo-WS-14 (ca. 9:1). Unfortunately the reaction is not instantaneous as it is at -5°C and if quenched after several hours overall conversion to products is only 30%. If the mixture is allowed to warm to room temperature the product ratios are identical to that at -5°C. Addition in refluxing THF increases the yield (81%) and the relative amount of neo isomer (2:1)[3].

The studies directed towards developing preparative HPLC isolations of key components in complex natural products continues. Although we had in the past easily isolated such materials menthol and neomenthol from peppermint oils, the isolation of various other components from browning mixtures or tobacco extracts have proven more difficult. These latter materials will undoubtebly still require preliminary simplification.[4]

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